

CLAIMS

What is claimed is:

1. A process for the manufacture of polytrimethylene ether glycol comprising the steps of:

5 a) providing (1) 1,3-propanediol reactant selected from the group consisting of 1,3-propanediol and/or oligomers or prepolymers of 1,3-propanediol having a degree of polymerization of 2-9 and mixtures thereof, and (2) a polycondensation catalyst; and

10 b) polycondensing the 1,3-propanediol reactant to form a polytrimethylene ether glycol at less than one atmosphere pressure.

2. The process of claim 1 wherein the 1,3-propanediol reactant is selected from the group consisting of 1,3-propanediol and/or dimer and trimer of 1,3-propanediol and mixtures thereof.

15 3. The process of claim 2 wherein the 1,3-propanediol reactant is selected from the group consisting of the 1,3-propanediol or the mixture containing at least 90 weight % of 1,3-propanediol.

4. The process of claim 2 wherein the 1,3-propanediol reactant is the 1,3-propanediol.

20 5. The process of claim 1 wherein polycondensation is carried out at a temperature of at least 150°C.

6. The process of claim 1 wherein the polycondensation temperature is no greater than 250°C.

7. The process of claim 1 wherein the polycondensation temperature is no greater than 210°C.

25 8. The process of claim 2 wherein the polycondensation temperature is no greater than 210°C.

9. The process of claim 7 carried out in batch mode.

10. The process of claim 1 wherein the polycondensing pressure is less than 500 mm Hg (66 kPa).

30 11. The process of claim 1 wherein the polycondensing pressure is less than 250 mm Hg (33 kPa).

12. The process of claim 1 wherein the polycondensing pressure is less than 100 mm Hg (13 kPa).

35 13. The process of claim 1 wherein the polycondensing pressure is less than 50 mm Hg (6.6 kPa).

14. The process of claim 1 wherein the polycondensing pressure is less than 5 mm Hg (660 Pa).

15. The process of claim 8 wherein the polycondensing pressure is less than 250 mm Hg (33 kPa).

16. The process of claim 1 wherein the polytrimethylene ether glycol number average molecular weight is greater than 1,000.

5 17. The process of claim 16 wherein the number average molecular weight is greater than 1,500.

18. The process of claim 16 wherein the number average molecular weight is greater than 1,650.

10 19. The process of claim 16 wherein the number average molecular weight is greater than 2,000.

20. The process of claim 1 wherein the polytrimethylene ether glycol number average molecular weight is less than 5,000.

21. The process of claim 20 wherein the number average molecular weight is less than 4,000.

15 22. The process of claim 20 wherein the number average molecular weight is less than 3,500.

23. The process of claim 18 wherein the number average molecular weight is less than 5,000.

20 24. The process of claim 18 wherein the number average molecular weight is 4,950 or less.

25. The process of claim 1 further comprising purifying the polytrimethylene ether glycol to a dispersity of 1.5 to 2.1.

26. The process of claim 1 wherein the resulting polytrimethylene ether glycol has an APHA color of less than 120.

25 27. The process of claim 26 wherein the APHA color is less than 100.

28. The process of claim 26 wherein the APHA color is less than 50.

29. The process of claim 1 further comprising purifying the polytrimethylene ether glycol to an unsaturation of less than 20 meq/kg.

30 30. The process of claim 29 wherein the unsaturation is less than 15 meq/kg.

31. The process of claim 1 wherein the resulting polytrimethylene ether glycol has a content of cyclic ether oligomers of less than 2%.

32. The process of claim 1 further comprising purifying the polytrimethylene ether glycol to a cyclic ether oligomer content less than 1%.

35 33. The process of claim 1 wherein the reaction mixture further comprises up to 50 mole %, based on all diols present, of a comonomer diol other than oligomers of 1,3-propanediol.

34. The process of claim 33 wherein the comonomer diol is selected from the group consisting of 2-methyl-1,3-propanediol, 2,2-dimethyl-1,3-propanediol, 2,2-diethyl-1,3-propanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, 1,12-dodecanediol and mixtures thereof.

5 35. The process of claim 33 wherein the comonomer is selected from the group consisting of 2-methyl-1,3-propanediol, 2,2-dimethyl-1,3-propanediol, and 2,2-diethyl-1,3-propanediol.

36. The process of claim 1 wherein the process is carried out with the 1,3-propanediol and it has a purity greater than 99%.

10 37. The process of claim 1 wherein the 1,3-propanediol and up to 10% of the low molecular weight oligomers.

38. The process of claim 1 wherein the polytrimethylene ether glycol has a number average molecular weight of 1,500 - 4,950 and an APHA color of less than 120, the pressure in the reduced pressure stage is less than 250 mm Hg (33 kPa) and the polycondensation temperature is 170 - 190°C.

15 39. A process for the manufacture of polytrimethylene ether glycol comprising the steps of:

- a) providing (1) 1,3-propanediol and (2) a polycondensation catalyst;
- 20 b) condensing 1,3-propanediol to form oligomer or prepolymer of 1,3-propanediol having a degree of polymerization of 2-9 or a mixture comprising one or more thereof; and
- c) polycondensing the oligomer or prepolymer of 1,3-propanediol having a degree of polymerization of 2-9 or a mixture comprising one or more thereof, to form a polytrimethylene ether glycol at less than one atmosphere pressure.

25 40. The process of claim 39 wherein step b) is carried out at about atmospheric pressure, the pressure in step c) is less than 300 mm Hg (40 kPa), the temperature in step b) is 150-210°C and the temperature in step c) is 170-250°C.

30 41. The process of claim 40 wherein the temperature in step b) is 170-210°C and the temperature in step c) is 180-210°C.

42. The process of claim 41 wherein in step b), 1,3-propanediol is condensed to dimer and trimer.

35 43. The process of claim 41 wherein the pressure for step c) is less than 250 mm Hg (33 kPa) and the polytrimethylene ether has a number average molecular weight of 1,650 to 4,950.

44. Polytrimethylene ether glycol produced by the process of claim 1.

45. The polytrimethylene ether glycol of claim 44 having a number average molecular weight of greater than 1,650.

46. Polytrimethylene ether glycol having a number average molecular weight greater than 1,500, an APHA color of less than 120, an unsaturation of less than 20 meq/kg, and a content of cyclic ether oligomers of less than 2%.

5 47. The polytrimethylene ether glycol of claim 46 having a dispersity greater than 1.5 and an alkalinity in the range of -2 to +1.

48. The polytrimethylene ether glycol of claim 47 having a number average molecular weight of from 1,650 to 4,000.

10 49. The polytrimethylene ether glycol of claim 48 having an APHA color of less than 100, an unsaturation of less than 15 meq/kg and a cyclic ether content of less than 1%.